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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/US93/03903 (22) International Filing Date: 27 April 1993 (27.04.93) (30) Priority data: 876,316 30 April 1992 (30.04.92) US (71) Applicant: J.M. HUBER CORPORATION [US/US]; Browns Dock & Navesink Roads, Rumson, NJ 07760 (US). (72) Inventors: PRESTON, Barry, W. ; 4022 Tabernacle Road, Whiteford, MD 21160 (US). KOSIN, John, A. ; 950-I Hillswood Road, Bel Air, MD 21160 (US). ANDREWS, Claude, R. ; 1810 Parkside Drive, Pasadena, MD 21122 (US).		(74) Agent: PRICE, Robert, L.; Lowe, Price, LeBlanc & Becker, 99 Canal Center Plaza, Suite 300, Alexandria, VA 22314 (US). (81) Designated States: AU, CA, FI, JP, NO, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: METHOD FOR PRODUCTION OF SYNTHETIC HYDROTALCITE (57) Abstract Methods for producing synthetic hydrotalcite having a purity level of 99 percent or greater and an average particle size of less than 2 microns. According to one embodiment, a <u>source of magnesium</u> , a carbonate source and a <u>source of aluminum</u> are combined and reacted together under hydrothermal conditions. According to another embodiment in which the resulting product has an average particle size of less than 1 micron, a source of magnesium and a carbonate source are reacted together to form a hydromagnesite which is then reacted with a source of aluminum under hydrothermal conditions.		

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METHOD FOR PRODUCTION OF SYNTHETIC HYDROTALCITERelated Applications

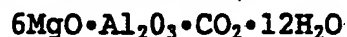
This application is a continuation-in-part application of U.S. Patent Application Serial No. 07/812,195, filed December 20, 1991 and U.S. Patent Application Serial No. 07/876,316, filed April 30, 1992.

Technical Field

The present invention relates to hydrotalcite and methods of producing synthetic hydrotalcite. More particularly, the present invention relates to methods of producing highly pure synthetic hydrotalcite which has a very small platelet morphology.

Background Art

Hydrotalcite is a naturally occurring mineral which is found in relatively small amounts in limited areas. Hydrotalcite is known to be useful as an antacid which has a neutralizing effect on gastric juices. In addition to natural hydrotalcite, synthetic hydrotalcites have been produced. U.S. Patent Nos. 3,539,306 and 3,650,704 to Kimura et al disclose synthetic hydrotalcites and methods for their manufacture. The synthetic hydrotalcite of these patents is described as having the following composition:



Hydrotalcites which have ² needlelike crystalline structures are described in U.S. Patent No. 4,351,814 to Miyata.

5 U.S. Patent No. 4,629,626 to Miyata et al discloses a hydrothermally treated product useful for treating an iron deficiency, which compound has a hydrotalcite like crystal structure and includes iron, magnesium, aluminum, and water within the structure.

10 U.S. Patent No. 4,904,457 to Misra discloses a method of producing a high purity hydrotalcite by reacting activated magnesia with a Bayer liquor at approximately 95°C.

15 U.S. Patent No. 4,883,533 to Kosin et al discloses a method of producing a modified synthetic hydrotalcite from a source of magnesium, a source of aluminum and a source of carbonate.

20 There remains a need for a method of producing hydrotalcite having a purity level of 99 percent or greater which has a very small particle size using inexpensive, commercially available reactants.

Disclosure of the Invention

25 It is accordingly one object of the present invention to provide a method of producing synthetic hydrotalcite from inexpensive, commercially available reactants.

Another object of the present invention is to provide a method of producing a synthetic hydrotalcite which has a purity level of 99 percent or greater, a uniform platelet morphology, and an average particle size of below 2 microns.

30 A further object of the present invention is to provide a method of producing a synthetic hydrotalcite which has a purity level of 99 percent or greater, a uniform platelet morphology, and an average particle size

of less than 0.5 microns³ with a particle size range of approximately 0.25 to 0.75 microns.

According to these and further objects of the present invention which will become apparent as the description thereof proceeds, the present invention provides for a method of producing synthetic hydrotalcite which involves:

reacting a mixture of a magnesium source, a carbonate source, alumina trihydrate, and an alkali metal aluminate under hydrothermal conditions at a temperature of about 160° - 200°C,

wherein the molar ratio of the alumina trihydrate to the alkali metal aluminate is between about 10:1 to 5:1,

the molar ratio of the magnesium source to the carbonate source is between about 0.9:1 to 1.1:1, and

the atomic ratio of the magnesium to total aluminum ion is between about 2.2:1 to 2.3:1

The present invention further provides a method of producing synthetic hydrotalcite which involves:

reacting an aqueous slurry of hydromagnesite with an aluminum source under hydrothermal conditions at a temperature of about 120° - 250°C for a period of about 0.1 - 1 hour, to produce a hydrotalcite,

wherein the aluminum source is selected from the group consisting of alkali metal aluminate, Bayer liquor, and a mixture of alkali metal aluminate and aluminum trihydrate, and

the molar ratio of magnesium to aluminum is between about 2.0 - 2.25

Description of the Invention

The present invention is directed to methods of producing synthetic hydrotalcite which utilize inexpensive reactants and which produce a synthetic hydrotalcite product 99 percent pure or greater. The

methods of the invention produce a synthetic hydrotalcite product which has an average particle size which is less than 2 microns, and in some cases less than 0.5 microns. The synthetic hydrotalcite produced by the methods of the present invention have a uniform platelet morphology.

The synthetic hydrotalcites which are produced by the methods of the present invention can be used in any known manner such as for fillers, reinforcing agents, or flame retardants, in a variety of polymer systems, including plastics and elastomers. In addition, due to the highly pure nature of the synthetic hydrotalcites, the present invention is particularly useful for producing synthetic hydrotalcites which are used in pharmaceutical applications such as antacids.

In general, the methods of producing synthetic hydrotalcites according to the present invention involve reacting a magnesium source, a carbonate and an aluminum source. However, it has been discovered during the course of the invention that two different reaction schemes are useful for producing the synthetic hydrotalcites of the present invention. One reaction scheme, which is hereafter referred to as the first embodiment of the present invention, involves a rapid reaction between the magnesium source, carbonate and aluminum source. This embodiment produces a synthetic hydrotalcite which is 99 percent pure or greater and has an average particle size of less than 2 microns, and typically less than 1 micron.

In another reaction scheme, which is hereafter referred to as the second embodiment of the invention, the magnesium source and the carbonate are first reacted together to form an intermediate product which is then reacted with the aluminum source. This embodiment produces a synthetic hydrotalcite which is 99 percent pure or greater and has an average particle size of less

than 0.5 microns and a particle size range of approximately 0.25 to 0.75 microns.

In addition to utilizing inexpensive reactants, each embodiment of the present invention provides particular advantages. For example, the first embodiment provides a simple, rapid method of producing highly pure synthetic hydrotalcite. The second embodiment provides a method of producing a synthetic hydrotalcite which has an extremely small, and relatively uniform particle size.

According to the first embodiment of the present invention, synthetic crystalline hydrotalcite is produced by combining together and reacting a mixture comprising a magnesium source, a carbonate, and an aluminum source under aqueous hydrothermal conditions at a temperature of about 160° - 200° C.

According to the second embodiment of the present invention, synthetic crystalline hydrotalcite is produced by combining together and reacting a source of magnesium and a source of carbonate under atmospheric conditions, preferably at a temperature between 60° - 80°C, to produce a hydromagnesite intermediate precipitate. Thereafter, the hydromagnesite intermediate, made as above or otherwise, is reacted with a source of aluminum under hydrothermal conditions at a temperature of about 120° - 250°C for approximately 0.1 - 1.0 hour to produce synthetic hydrotalcite.

In each embodiment of the present invention the magnesium source may be selected from the group consisting of magnesium oxide, magnesium hydroxide or mixtures thereof. The preferred source of magnesium is magnesium hydroxide which can be provided as aqueous slurry with a solids percent of about 40 to 60%. Equivalent sources of magnesium may be used.

The carbonate source may be selected from alkali metal bicarbonates, alkali metal carbonates, carbon dioxide gas, carbonic acid, or mixtures thereof.

The aluminum source may be Bayer liquor, alkali metal aluminate or a mixture of alkali metal aluminate and solid alumina trihydrate. It is noted that while a mixture of alkali metal aluminate and solid alumina trihydrate can be used, it has been discovered that alumina trihydrate alone does not give a product having the desired fine particle size.

According to the first embodiment of the present invention which is discussed below and through Example 4, the magnesium source, carbonate, and aluminum source reactants are combined together in a closed reactor in an aqueous medium to react under hydrothermal conditions. The reactant mixture is then heated at a temperature of about 160° - 200°C for about 1 to 3 hours. During the course of the present invention, the inventors discovered that at temperatures below about 160°C, the reaction proceeds very slowly, if at all, and that at temperatures above about 200°C, dawsonite contaminant is produced. The more preferred temperature range has been determined to be 170° - 190°C, with a temperature of 175°C being the most preferred.

After the reaction, the product can be isolated by filtration and dried at an elevated temperature, e.g., 105°C according to conventional processes. It has been found that the hydrotalcite produced by this embodiment of the present invention is easy to filter and the dried product is very friable. As discussed above, the resulting synthetic hydrotalcite has an average particle size less than about 2 microns, and typically, the resulting synthetic hydrotalcite produced by this embodiment has an average particle size of less than 1 micron. The synthetic hydrotalcite product has a platelet morphology and is produced at a purity level of 99 percent or greater, with a typical purity of 100 percent.

During the course of the present invention, it has been determined that, for this embodiment, it is critical that the aluminum be present as a mixture of alkali metal aluminate and solid alumina trihydrate. Preferably, the alkali metal aluminate comprises sodium aluminate. It has also been determined that there is a proper ratio of the alkali metal aluminate and solid alumina trihydrate which produces the unique synthetic hydrotalcite of the present invention and distinguishes the method of the present invention. In this regard, the present inventors have discovered that the molar ratio of the alumina trihydrate to the alkali metal aluminate must be between about 10:1 to 5:1, with a ratio of 9 to 7:1 being preferred. A more preferred ratio of alumina trihydrate to alkali metal aluminate has been determined to be 8:1.

It has further been determined that it is important that the molar ratio of the alkali metal aluminate, which is preferably sodium aluminate ($\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$), be as low as possible, i.e. have a low alkali metal content on a molar basis. During the course of the present invention it has been discovered that as the molar ratio of the alkali metal aluminate increases above 1.25, the possibility of producing dawsonite, as an impurity, along with the hydrotalcite increases. When the molar ratio of the alkali metal aluminate was as high as 1.5, a significant amount of dawsonite contaminant was produced with the hydrotalcite. Accordingly, for purposes of the present invention, the preferred alkali metal aluminate is a sodium aluminate, which is commercially available at a molar ratio of 1.25 $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$. However, it is to be understood that a sodium aluminate having a molar ratio of less than 1.25 would be more preferred.

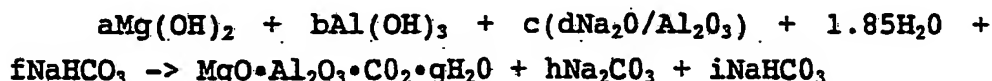
In addition to avoiding the production of dawsonite contaminant which is undesirable, it has been discovered that excess sodium also results in unfavorable economics and results in a product which is more difficult to wash.

It is possible to use a Bayer liquor as a source of aluminum with some sodium carbonate.

The carbonate reactant used in the first embodiment of the present invention is preferably present as an alkali metal bicarbonate. The use of carbon dioxide and mixtures of an alkali metal bicarbonate is less preferred in this embodiment of the invention, but could be used. The preferred alkali metal bicarbonate is sodium bicarbonate.

During the course of the present invention, it has been discovered that the ratio of the magnesium source to the bicarbonate source has to be carefully controlled. That is, this ratio must be kept in the range of substantially 0.9:1 to 1.1:1. If the ratio of magnesium to bicarbonate is less than 0.9, dawsonite contaminant is produced. If this ratio is greater than 1.1, the purity of the synthetic hydrotalcite decreases.

In the first embodiment of the present invention, it has been discovered that this novel reaction proceeds according to the following molar ratios using the preferred reactants:



wherein a equals 4.4 to 4.6; $d < 1.5$; $b/c = 5$ to 10; $a/f = 0.9$ to 1.1; and h and i are positive numbers.

It is noted that the atomic ratio of magnesium to total aluminum ion from all sources is substantially 2.25:1, i.e. about 2.2:1 to about 2.3:1.

The hydrotalcite produced according to the first embodiment of the present invention has a high purity and a small particle size. The hydrotalcite product has been determined to be suitable for use as a stabilizer and acid acceptor in polymer systems such as polypropylene, polyvinyl chloride, chlorosulfonated polyethylene, etc.

The following examples are presented to illustrate features and characteristics of the first embodiment of the present invention which is not to be considered as limited thereto. In the examples and throughout the specification, percentages are by weight unless otherwise indicated.

Comparative Example No. 1

in this example a total of 1,594 grams of water, 250 grams of a $Mg(OH)_2$ in slurry (55% solids), 240 grams of sodium aluminate solution (molar ratio of $2.0Na_2O/Al_2O_3$) and 204 grams of $NaHCO_3$ were placed in a stirred one-gallon Parr reactor. The reactor was heated to $175^\circ C$ and stirred for 2 hours. The product was isolated by filtration and dried at $105^\circ C$. The resulting hydrotalcite was found to be 98% pure by x-ray diffraction analysis (XRD). The molar ratios of the reactants were as follows:

<u>REACTANTS</u>	<u>MOLAR RATIO</u>
$Mg(OH)_2$	5.04
Al_2O_3	1.00
CO_2	5.13

It was found that on a small laboratory scale, the purity of the hydrotalcite produced according to the method of this example was approximately 98 percent. As this method is scaled up to commercial capacity the purity may drop as low as 95 percent due to physical mixing limitations.

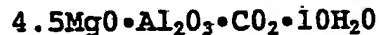
Comparative Example No. 2

In this example a total of 2,475 grams of water, 391.4 grams of $Mg(OH)_2$ slurry (55% solids), 128.7 grams of alumina trihydrate solid (average particle size 2 microns) and 188.1 grams of $NaHCO_3$ were placed in a

stirred one-gallon Parr reactor. The reactor was heated to 175°C and stirred for 2 hours. The hydrotalcite product was isolated by filtration and dried at 105°C. The resulting hydrotalcite had a platelet morphology and an average particle size of 2 to 3 microns with a purity determined by x-ray diffraction of about 95 percent.

It was found that when 100 percent of the aluminum comes from 1 micron solid alumina trihydrate (ATH) and when the method of this example is used, the product purity level approaches 100 percent. However, the particle size of the product was always greater than 2 microns and often ranged up to 4 or 5 microns. If this process were scaled up for production purposes, the size of the particles would also increase due to mixing limitations.

A chemical analysis of the synthesized hydrotalcite produced by this example was performed and the following empirical formula was derived:



Example 3

In this example a total of 1,125 grams of H₂O was added to a 2-liter Parr pressure reactor followed by 141 grams of NaHCO₃, 192 grams of Mg(OH)₂ in slurry (51% solids), 46.8 grams of alumina trihydrate (average particle size 1 - 2 microns) and 30.6 grams of sodium aluminate solution (molar ratio 1.25Na₂O/Al₂O₃). The reaction slurry was continuously agitated, heated to 175°C and allowed to react for 2 hours. After the reaction, the hydrotalcite product was isolated by filtration and washed with 500 ml. of hot water. The resultant wet cake was dried at 105°C overnight and then hammer milled.

The hydrotalcite product produced by this method had an impurity level of 100 percent (determined by x-ray

diffraction) and a platlet morphology with 77 percent of the platlets falling between 0.5 and 1.5 microns by Coulter counter analysis. The product had an average particle size of about 1 micron. The molar ratio of $\text{Al}(\text{OH})_3$ to sodium aluminate was 8 to 1. The molar ratio of the magnesium source to the bicarbonate source was 0.98:1 and the atomic ratio of magnesium source to the total aluminum ion from all sources was 2.25:1.

The purity of hydrotalcite is very important in determining its functionality in acid acceptor/stabilizing applications. As an example of such applications, hydrotalcite can be added to polyvinyl chloride formulations to function as an acid acceptor and thus enhance thermal stability. Hydrotalcite is known to react with HCl generated as the polyvinyl chloride begins to degrade to form the insoluble chloride form of hydrotalcite. A trace amount (2 to 5 percent) of magnesium hydroxide contaminant reacts to form the soluble magnesium chloride. Soluble chloride can cause yellowing of the PVC and aid in early degradation.

Example 4

In this example a hydrotalcite containing 2 percent MgOH_2 was added (0.5 phr) to a PVC formulation and compared with the hydrotalcite of 100 percent purity of the present invention. The samples containing the hydrotalcites were oven aged at 190°C and the samples were removed and examined every 5 minutes. It was found that after approximately 40 minutes the PVC containing 98 percent purity hydrotalcite changed from a light yellow color to black signifying degradation. The PVC sample containing 100 percent purity hydrotalcite did not degrade until after approximately 60 minutes. Accordingly, it can be seen that the performance characteristics of a 98 percent pure hydrotalcite vs. a hydrotalcite which is 100 percent pure is significant.

According to the second embodiment of the present invention, crystalline hydrotalcite is produced by first combining together and reacting a source of magnesium and a source of carbonate under atmospheric conditions, preferably at a temperature between 60° - 80°C, to produce a hydromagnesite intermediate precipitant. Thereafter, the hydromagnesite intermediate is reacted with a source of aluminum under hydrothermal conditions at a temperature of about 120° - 250°C for approximately 0.1 - 1.0 hour to produce hydrotalcite.

The reaction of the magnesium source and the carbonate source produces a pure hydromagnesite precipitant having no extraneous salts when CO₂ is used as the carbonate source. Although the CO₂ will react with the magnesium source at any temperature between 0° - 100°C, a water temperature of about 25° - 95°C is preferred for most efficient use of the carbon dioxide, with a temperature of 40° - 80°C being most preferred. If other carbonate sources are used, any extraneous salts should be washed from the hydromagnesite if desired before its introduction into the next step.

After formation of the hydromagnesite, an aluminum source is added to the reaction slurry so that the molar ratio of magnesium to aluminum is approximately between 2 to 2.25. This molar ratio is necessary in order to obtain the high purity of the final product. The aluminum source may be Bayer liquor, alkali metal aluminate or a mixture of alkali metal aluminate and solid alumina trihydrate. It is noted that while a mixture of alkali metal aluminate and solid alumina trihydrate can be used, it has been discovered that alumina trihydrate alone does not give a product having the desired fine particle size.

The preferred aluminum source is sodium aluminate which is commercially available at a molar ratio of 1.25 Na₂O/Al₂O₃. This aluminum source is preferred in order to

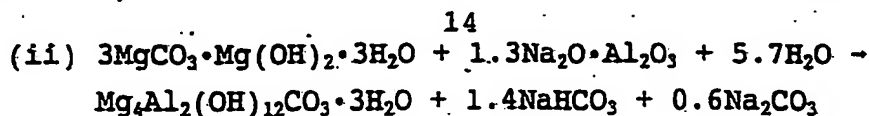
reduce the amount of excess sodium produced in the reaction. Excess sodium should be avoided if possible because it can result in unfavorable economics and can make the product more difficult to wash.

5 The hydromagnesite intermediate is placed together in a closed reactor in an aqueous medium with the aluminum component to react under hydrothermal conditions. This mixture is heated to a temperature of about 120° - 250°C under agitation for about 0.1 - 1.0
10 hour. The reaction temperature is not critical to the formation of the final product. However, if the reaction temperature is below about 120°C, the reaction proceeds more slowly. On the other hand, no benefit is gained by conducting the reaction at temperatures greater than
15 250°C. The more preferred temperature range for the reaction is about 140° - 180°C, with a temperature of 175°C being most preferred for fast reaction and high reactor throughput. A temperature of 140°C is more preferred for reduced energy demand.

20 After the reaction, the product is isolated by filtration and dried at an elevated temperature, e.g., 105°C. The resulting hydrotalcite is easy to filter and the dried product is very friable. The resulting hydrotalcite product has an average particle size less
25 than about 1 micron, and typically has an average particle size of 0.4 microns. The hydrotalcite product has a particle size which ranges from 0.25 to 0.75 micron. The particles produced have a uniform platelet morphology and a purity level of 99 percent or greater,
30 with a typical purity level of 100 percent.

In the second embodiment of the present invention, it has been discovered that the novel reaction proceeds according to the following equations using the preferred reactants:

35 (i) $4\text{Mg}(\text{OH})_2 + 3\text{CO}_2 \rightarrow 3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ and then



The hydrotalcite produced by this embodiment of the present invention has a high purity level and a small particle size which makes it particularly suitable for use as a stabilizer and acid acceptor in polymer systems such as polyethylene, polyvinyl chloride, chlorosulfonated polyethylene, etc.

If desired, the hydrotalcite of either embodiment of the present invention may be surface treated with a conventional anionic surface active agent, such as a higher fatty acid or its alkali metal salt, to aid its dispersibility in polymer applications. This surface treatment may be added at the beginning of the hydrothermal step, to the reacted slurry or to the final dried product. A more thorough discussion of the surface treatment of hydrotalcite may be found in U.S. Patent 4,558,102 to Miyata. The amount of surface treatment may vary as desired but it is preferred that the amount be sufficient to substantially coat the hydrotalcite product with at least a monolayer of treatment chemical.

The following example is presented to illustrate the novel nature of the second embodiment of the present invention which is not to be considered as limited thereto. In the example and throughout the specification, parts are by weight unless otherwise indicated.

Example 5

In this example a charge of 3.7 liters of water was added to a stainless steel baffled reactor with constant agitation. Agitation was maintained throughout the reaction process. The water was heated to 75°C and maintained during the reaction. In a separate addition vessel, 600 grams of a magnesium hydroxide slurry were

added and kept in continuous agitation. Carbon dioxide gas was injected into the water charge at a flow rate of about 0.9 liters per minute. Thereafter, the magnesium hydroxide slurry was added to the water charge at a rate of 5.2 grams per minute. The CO₂ flow rate was adjusted to maintain a reaction pH of 7.0 ± 0.3 throughout the reaction. Magnesium hydroxide slurry was added for exactly 90 minutes and a total of 468 grams was reacted in this time period. After ninety minutes, the magnesium hydroxide and CO₂ were stopped and the batch was allowed to digest 15 minutes with agitation. After digestion, the pH had increased to approximately 8.0 and accordingly CO₂ was again injected to bring the reaction slurry back down to pH 7.0 ± 0.3. The CO₂ was then stopped and 427 grams of sodium aluminate (18.9 percent Na₂O, 24.9 percent Al₂O₃, Na₂O/Al₂O₃ molar ratio equal 1.30) was added. The reactants were mixed for ten minutes. Thereafter, 2,700 grams of this reaction slurry were transferred to a one-gallon pressure reactor. The reactor was brought to a temperature of 175°C (120 psig steam) and the slurry was reacted for sixty minutes. It is presently believed that this reaction goes to completion within about 0.1 hour. The reaction product was isolated by filtration and washed with one liter of hot water. The resulting wet cake was dried at 105°C overnight. The dried wet cake was milled and submitted to x-ray diffraction analysis. The high purity of the reaction product (100%) was indicated by the absence of any other significant diffraction lines.

A scanning electron photomicrograph showed a hydrotalcite with a platelet morphology with platelets ranging between 0.25 - 0.75 microns in diameter. Sedigraph analysis showed the hydrotalcite to have an average particle size of 0.4 microns. A chemical analysis showed the following empirical formula

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CLAIMS

1. A method of producing synthetic hydrotalcite which comprises:

reacting a mixture of a magnesium source, a carbonate source, alumina trihydrate, and an alkali metal aluminate under hydrothermal conditions at a temperature of about 160° - 200°C,

wherein the molar ratio of the alumina trihydrate to the alkali metal aluminate is between about 10:1 to 5:1,

the molar ratio of the magnesium source to the carbonate source is between about 0.9:1 to 1.1:1, and

the atomic ratio of the magnesium to total aluminum ion is between about 2.2:1 to 2.3:1.

2. The method of Claim 1 wherein the molar ratio of the alumina trihydrate to the alkali metal aluminate is approximately 9-7:1.

3. The method of Claim 1, wherein the molar ratio of the aluminum trihydrate to alkali metal aluminate is about 8:1.

4. The method of Claim 1, wherein said magnesium source is magnesium hydroxide, said carbonate source is sodium bicarbonate, said alkali metal aluminate is sodium aluminate and the molar ratio of the aluminum trihydrate to sodium aluminate is about 8:1, and the molar ratio of the magnesium hydroxide to sodium bicarbonate is between about 0.95:1 to 1.05:1.

5. The method of Claim 1, wherein said sodium aluminate has a molar ratio of 1.25

6. A method of producing synthetic hydrotalcite which comprises:

reacting an aqueous slurry of hydromagnesite with an aluminum source under hydrothermal conditions at a temperature of about 120° - 250°C for a period of about 0.1 - 1 h ur, to produce a hydrotalcite,

5 wherein said aluminum source is selected from the group consisting of an alkali metal aluminate, Bayer liquor, and a mixture of an alkali metal aluminate and aluminum trihydrate, and

10 the molar ratio of magnesium to aluminum is between about 2.0 - 2.25.

7. The method of Claim 6, wherein the hydromagnesite slurry is made by:

5 reacting an aqueous slurry of a magnesium source with a carbonate source at ambient pressure and at a temperature between about 25° - 95°C,

wherein said magnesium source is selected from the group consisting of magnesium hydroxide, magnesium oxide and mixtures thereof, and

10 the carbonate source is selected from the group consisting of alkali metal bicarbonate, alkali metal carbonate, carbon dioxide, carbonic acid and mixtures thereof.

8. The method of Claim 6, wherein said aluminum source is sodium aluminate

9. The method of Claim 6, wherein said sodium aluminate has a molar ratio of 1.25

10. A synthetic hydrotalcite product produced by the method of claim 1.

11. A synthetic hydrotalcite product produced by the method of claim 6.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US93/03903

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : C01F 1/00, 5/24, 7/02; C01B 31/24

US CL : 423/115, 129, 419P, 430, 432, 600, 630

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/686; 423/115, 129, 419P, 430, 432, 600, 630

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS, Hydrotalcite, hydromagnesite, magnesium hydroxide, magnesium oxide, bicarbonate, CO, carbondioxide, carbonic acid.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 3,539,306 (Kumura et al) 10 November 1970, see col. 2, line 39 to col. 4, line 10, col. 3, lines 17 and 42-43, col. 2, lines 44-46.	1-11
Y	US, A, 3,447,899 (Maskal et al) 03 June 1969, see col. 1, line 58 to col. 2, line 63.	1-11
Y X	US, A, 4,883,533 (Kosin et al) 28 November 1989, see col. 2, line 67 to col. 3, line 27 and the abstract.	1-9 10,11

☐ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be part of particular relevance "E" earlier document published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed		"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "Z" document member of the same patent family
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Date of the actual completion of the international search

23 JUNE 1993

Date of mailing of the international search report

05 AUG 1993

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